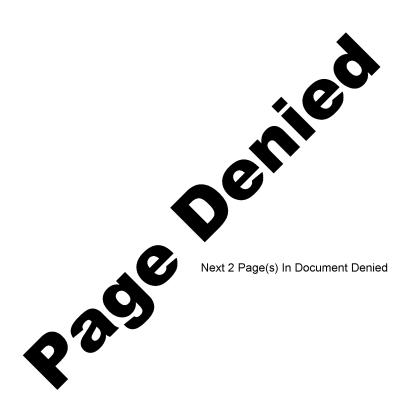
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THE INFLUENCE OF CRYSTAL PROPERTIES AND GRAIN SUBSTRUCTURE ON HARDNESS I. Fe-Ni AND Fe-Si ALLOYS*

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An investigation has been made of the temperature dependence of yield point and hardness in alloys in the annealed and strain-hardened states. Analysis of the experimental data confirms previous conclusions that the resistance of a strain-hardened material to deformation is determined by two factors: the crystal properties of the material (resistance to the movement of dislocations inside the regions of a crystal which are free of subboundaries) and granular substructure (the size of submicroregions, presence of internal boundaries in the grain, degree of disorientation of the separate regions). On the basis of the additivity of the effect of both vectors calculation has been made of the temperature dependence of the hardness of a strain-hardened alloy and it has been compared with experimental data. A relationship has been established between the magnitude of type II distortions and temperature dependence of $\sigma_{\mathcal{S}}$ and HV.

In investigations of binary alloys of iron it has been established that after the same degree of cold deformation (80%) strength will be greater in an alloy which has undergone a high degree of second-grade distortion (microstresses). After strengthening, the size of the regions of coherent scattering (blocks) was practically the same for all the alloys, being 300-400 Å [1]. Similar laws have been est blished already in the investigation of the fine structure and mechanical properties of quenched steels with different carbon contents [2, 3].

From an analysis of the experimental material the suggestion can be put forward [1, 3, 4], that the actual second-type distortions which arise on strengthening are not a major factor in increasing the resistance of a material to plastic deformation. The accuracy of this proposition was then confirmed experimentally in papers [5-7, 9]. The conclusion was drawn that the most important crystallostructural factors responsible for the strengthening of metals and alloys is the break-up of the grain into fragments 10^{-3} and 10^{-4} cm in size with considerable disorientation among the fragments and the formation of internal submicroscopic regions of coherent scattering of X-rays [1, 3, 7].

The direct connexion between the strength characteristics (hardness, yield point) of strain-hardened alloys and the extent of second-type distortions can be attributed not the presence of these considerable distortions but to the fact that the crystals of these alloys have non-uniform properties, even in the annealed state. Here one is referring to those properties which determine the resistance of nonstrain-hardened materials to the passage of elementary acts of plastic deformation (resistance to the movement of dislocations in the sector free of sub-boundaries). The extent of the second-type distortions in a strain-hardened alloy itself is determined by this resistance and is itself only an indicator. It can be regarded as a measure of the limit of elastic deformation in the microregions of the material in question. Consequently, it is not only the yield point of annealed alloys but also the degree of second-type distortions ($\Delta a/a$) which must be used as the characteristics of the individual strength properties of crystals. Thus the absolute value of the strength properties of alloys in the strain-hardened state depend not only on the appearance of a fine crystalline grain structure but also on the properties of the metal crystals themselves in the initial as-annealed state.

In papers [1-3, 5] a different level of hardness was achieved in iron alloys by varying the

^{*} Fiz. metal. metalloved., 11, No. 4, 609-614, 1961.

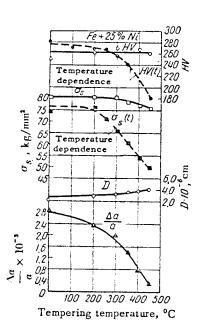


FIG. 1. Dependence of hardness HV, yield point σ_s , size of the fields of coherent scattering D on tempering temperature for the quenched alloy Fe + 25 % Ni; temperature dependence of yield point σ_s (t) and hardness HV (t).

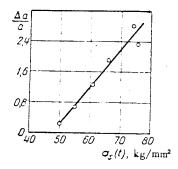


FIG. 2. Relation between second-type distortions $\Delta a/a$ and yield point for the alloy Fe + 25 % Ni.

properties of the crystals by alloying the iron with various different elements or changing the concentration of one of them. It is, however, possible to obtain different properties in the same material without change of chemical composition, by varying the temperature. The property which interests us, yield point, is known to be dependent on temperature. Therefore, if the strength characteristics of a strain-hardened metal are measured at various different temperatures, they should vary with the

temperature parallel to the variation in the yield point of the annealed metal as long as the substructure of the grain remains unchanged with temperature variation. The extent of the second-type distortions which occur as a result of deformation at various different temperatures should vary in exactly the same way. Besides this there should be a definite relationship between the $\Delta a/a$ value and yield point at the various different stages of softening. Of course, the beginning of the reduction in secondtype distortions on heating will occur at the temperature at which the elastic deformation limit of the crystals becomes less than the residual elastic deformation of the microregions which arise after strengthening. At this temperature the crystals are not in a position to withstand such a degree of microstresses as arises after deformation at lower temperatures. Stress relaxation then occurs.

Consequently the extent of second-grade distortions determined after the specimens have been heated up to various different temperatures should serve as a characteristic of the maximum elastoplastic deformation which the crystal of the material can maintain at the temperature in question. The $\Delta a/a$ curve after heating up to various temperatures (determined at 20°C) should reflect the temperature dependence of the elastic limit of the material.

It is the aim of the present work to study the influence of variation of crystal properties due to temperature changes, on the strength characteristics of metals after strain-hardening and on the extent of elastic deformations in microregions (second-type distortions).

The binary alloys Fe + 25% Ni and Fe + 1.15% Si were used for the investigation. The first was strengthened by quenching and the other by cold rolling with a total reduction of 50%. The reason for choosing the alloy Fe + 25% Ni as an object for investigation was the fact that when it is tempered to 450°C almost complete relief of second-type distortions is observed with practically no change in the size of the regions of coherent scattering [6].

The principal methods used for the investigations were those of X-ray diffraction analysis based on the study of the width of interference lines. The width of interference lines (110) and (220) were determined. The X-ray photographs were made in $\operatorname{Fe} K_a$ radiation. The magnitude of the type II distortions and the size of the regions of coherent scattering [8] were determined from the broadening effect of

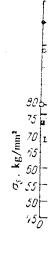


FIG. 3. Tempe yield p

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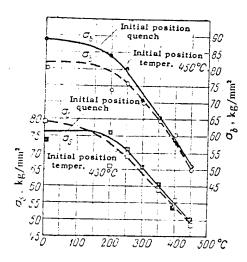


FIG. 3. Temperature dependence of UTS (σ_{R} (t)) and yield point for the alloy Fe + 25 % Ni.

the interference lines. The following mechanical characteristics were determined:

(1) yield point (σ_s) , UTS (σ_B) and Vickers hardness (HV) after strain-hardening and at various stages of softening;

(2) temperature dependence of σ_s , σ_B and HV which, in that work, were referred to as σ_s (t), σ_B (t), HV (t) respectively, in two states: after strengthening and after annealing.

Fig. 1 shows the variations after heating of the fine structure characteristics $(\Delta a/a, D)$ the temperature dependence of yield point σ_s (t) and of hardness HV (t) for the binary alloy Fe + 25% Ni, previously strain-hardened by quenching. For comparison the same illustration shows the σ_s and HV values measured at 20° after specimens had been tempered at various different temperatures.

The temperature dependence characteristics of yield point are very different from the changes in σ_s , HV measured at 20°C. There is a difference of 25 kg/mm² in yield point measured at 450°C and that measured at 20°C in a specimen which had been tempered at the same temperature. The corresponding variation in hardness is 80-90 units. The course of the variation in $\Delta a/a$, σ_s (t) and HV (t) is practically the same. This is confirmed by Fig. 2. The fact that there is practically no change in the yield point of specimens tempered at various different temperatures when this is measured at 20°C should mean that in these experiments the nature of the change in yield point reflects in the main, the nature of the

change in the substructure on heating. Actually, in the alloy Fe + 25% Ni investigated, the substructure of specimens heated to 450°C remains practically unchanged, the D value remains the same. This is also true of yield point.

The following experiments were carried out on the same alloy. After strengthening by quenching the specimens were tempered at 430°C to relieve the greater part of the second-type stresses. The temperature dependence of the yield point of specimens which had previously been tempered at 430°C was found to coincide with the σ_s (t) dependence of non-tempered ones. These data are in agreement with conclusions expressed earlier that the presence of second-type distortions does not in itself cause an increase in the resistance of the metal to deformation.

The temperature dependence of UTS σ_B (t) of alloys which have been quenched and first tempered (Fig. 3) has the same form as that of σ_s (t). The slight divergence of the σ_s (t) and σ_B (t), in the tempered and non-tempered specimens appears to be due to the small variations in the size of blocks which occur as a result of an hour's tempering at 430°C.

The alloy Fe + 25 % Ni is particularly suitable as an object of investigation. A number of experiments can be carried out on it which, in our opinion, make particularly clear the role of second-type distortions in strength hardening [6] and the connexion between $\Delta a/a$ variations after various different heating temperatures and the temperature dependence of yield point. This alloy has however, the disadvantage that it is not possible to bring about softening to any degree by heating, due to the rather low temperature for the commencement of the reverse $\alpha \rightarrow \gamma$ transformation. Austenite which is formed at above 460°C transforms to martensite even with very slow cooling. It was not herefore possible to establish on this alloy whether there was any connexion between $\Delta a/a$ and σ_s (t) in the annealed specimen.

Experiments similar to those carried out on the binary alloy Fe + 25% Ni were also done on the alloy Fe + 1.15% Si. After the iron silicon alloy had been strengthened second-type distortions $\Delta a/a = 1.75 \times 10^{-3}$, arose which did not vary when heated to 300°C. Reduction in the $\Delta a/a$ value takes place at much higher temperatures; after heating at 500-600°C it is 0.5×10^{-3} . The regions of coherent scattering start to grow at exactly the

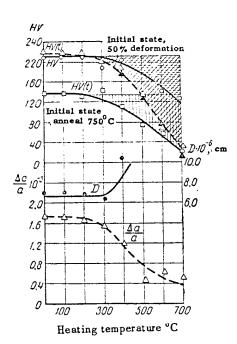


FIG. 4. Variation in the hardness characteristics and structure on heating in the alloy Fe + 1.15 % Si:

- o hardness (HV) of deformed alloy at 20° after heating to various different temperatures;
- □ hardness HV (t) of annealed alloy at various different temperatures;
- Δ hardness of deformed alloy at various different temperatures, found experimentally (upper curve);
- A calculated values for the hardness of deformed

same temperatures as the second-type distortions start to decrease (Fig. 4).

After comparing the data for the change in hardness of deformed specimens measured at 20°C after heating at various different temperatures, with the temperature dependence of hardness, we can see that there is no change in the HV and HV (t) values when heated to 300°C and they coincide. The change in HV (t) is greater at higher temperatures than in HV. This difference becomes more apparent as the temperature increases and at 700° it is a 100 Vickers unit.

Determination was also made of the temperature dependence of the hardness of specimens first annealed at 750°C. There was little change in the hardness of annealed specimens when heated to 300°C and a sharp drop from 135 to 25 HV in the 350-700°C. The temperature dependence of the hardness of previously annealed specimens indicates the nature of the reduction in the resistance to deformation due to change in the properties of

the crystals with temperature. In this range of temperatures the structure of the specimens should not undergo any kind of change (as they were previously heated to 750°) and therefore it should not

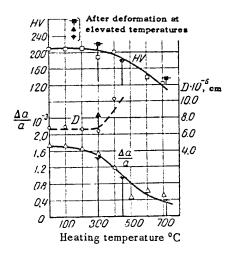


FIG. 5. Hardness, second-type distortions, size of regions of coherent scattering in alloy Fe + 1.15% Si deformed at 20°, after heating to various different temperatures (unfilled squares, triangles, circles); the same for alloy deformed at various different temperatures (filled squares, triangles, circles).

be dependent on HV changes.

The curve for the variation in HV measured at 20°C, in deformed specimens heated to various different temperatures, does on the other hand only reflect the changes in the micro - and submicrostructure of the grain which occur as the temperature is increased. If the temperature dependence of the hardness of strain-hardened specimens is measured, it will be found that at each temperature changes in the substructure of the grain and the properties of the crystals also have an effect on hardness.

On this basis we plotted a "theoretical" curve for the temperature dependence of hardness HV (t) for cold deformed alloy. We used the curve for the temperature dependence of the hardness of an annealed alloy and that for the variation in hardness measured at room temperature (HV) after heating a strain-hardened alloy to different temperatures. The reduction in hardness at each temperature due to change in the properties of the crystals can be

found from reduction in substructur tent of the The results and indicat obtained ar obtained ex

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found from the first curve, and from the second, the reduction in hardness due to coarsening of the grain substructure. Put together they should give the extent of the decrease in hardness of the heated alloy. The results of the calculation are given in Fig. 4 and indicated by the black triangles. The values obtained are in very good agreement with the curve obtained experimentally (unfilled triangles).

If it is true that resistance to deformation at any given temperature is determined by the properties of the crystal of the material and the nature of the grain substructure, then the resistance to plastic deformation at elevated temperatures should be equal or less than the resistance of specimens strain-hardened at room temperature and tempered at various different temperatures. The $\Delta a/a$ value after deformation at elevated temperatures should be less than or equal to the $\Delta a/a$ value obtained after heating specimens deformed at room temperature to a corresponding temperature.

These experiments were carried out on Fe-Si specimens which had been deformed at 330, 480 and 750°C. When they were rolled under cold rolls their temperature was slightly reduced, by 40-60°C. The results were compared with data regarding the changes in $\Delta a/a$, D and HV in dependence on tempering temperature of specimens previously strainhardened at room temperature (Fig. 5).

The experimental data obtained are in very good agreement with the hypotheses put forward.

CONCLUSIONS

Second-type distortions, which reflect the properties of crystals, vary on heating parallel with the temperature dependence of the yield point of an annealed or strain-hardened material, so long as the substructure remained unchanged in the temperature range investigated. The change in yield point or hardness observed at 20°C in strain-hardened specimens heated to various different temperatures, is the result of a change in substructure (softening) which has occurred during heating. The strength of metals and alloys strain-hardened at various different temperatures is determined by the properties of the crystals at the temperatures in question and by the nature of the micro- and submicrostructure of the grain which arises upon this.

Translated by V. Alfard

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THE INFLUENCE OF CRYSTAL PROPERTIES AND GRAIN SUBSTRUCTURE ON HARDNESS II. IRON AND NICKEL*

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The width of X-ray diffraction lines and the hardness of polycrystalline specimens in the annealed and strain-hardened states have been measured at 20 and - 180°C. Strain-hardening was achieved by plastic deformation at both temperatures.

Variation of tempering temperature causes the same degree of change in the hardness of strain-hardened and annealed materials. The difference in the hardness of strain-hardened and annealed metals remains the same at both temperatures; it increases with the extent of fragmentation of the substructure of the grain.

The experimental results illustrate the additivity of the effect of the two factors on strength; crystal properties and grain substructure.

The resistance of a strain-hardened material to deformation is determined by the individual properties of the crystals and the changes in the micro- and substructure of the grain which occur on the strain-hardening [1-4]. The yield point of the initial material and the magnitude of second-type distortions (elastic limit of the microregions) of the material strain-hardened to saturation, may be used as the characteristics of the crystal properties. According to experimental data [5], the yield point of an unstrengthened material is defined by the resistance which encounters a sliding dislocation on a surface free of dislocation.

Crystal properties vary considerably with the degree of alloying and temperature. Yield point (σ_s) is known to be highly dependent on temperature below 20°C in metals with a body-centred cubic lattice or solid solutions on their base. In metals with a face-centred cubic lattice σ_s hardly varies at all when the temperature is reduced right down to -200°C. To assess the role of crystal properties and the substructure arising during strenghtening, on the increase in the resistance of a material to plastic deformation it seemed to us that it would be interesting to investigate the fine structure and mechanical properties of metals which had been

strengthened at temperatures below + 20°C. Iron and nickel were selected for the investigation as these metals are very different in the nature of the temperature dependence of their yield point below 20°C. The strain-hardening was achieved by compression in a 100-ton press at 20 and -180°C.

The main method of examination used was X-ray diffraction analysis based on a study of the width of interference lines; X-ray photographs were made in FeK radiation at +20 and -180° C*. Vickers hardness (HV) was determined at +20 and -180° C. After 1 hr anneal at 700° C the iron had a hardness of 65 HV at 20° C and the width of the interference line (220) was 11.0×10^{-3} rad. When the temperature of the annealed specimen was reduced to -180° C hardness increased from 65 to 185 HV. At the same time there was practically no change in the width of the interference line measured at low temperature, it was B (220) = 11.6×10^{-3} rad.

The iron specimen then underwent 30 per cent deformation at -180° C. Hardness increased from 185 to 220 HV and the width of the line (220) increased from 11.6 to 31×10^{-3} rad. After the specimen had been heated from -180 to $+20^{\circ}$ the width of the line reduced from 31 to 22×10^{-3} rad

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^{*} Fiz. metal. metalloved., 11, No. 4, 615-619, 1961.

^{*} Deformation, during which the temperature of the specimens increased by 15-20°C, was carried out in liquid nitrogen.

and hardness from 222 to 98 HV. After a second cooling to - 180° hardness measured at the same temperature, again increased to 220 HV and the width of the line remained the same as at + 20°C (Figs. 1, 2).

The increase of 120 units in the hardness of the annealed specimen when cooled to — 180°C can as a whole be attributed to the change in crystal properties with reduction in temperature. No structural changes should occur. This is confirmed by the measurements of the width of the interference lines. The increase in hardness from 185 to 222 HV after strain-hardening at — 180° is determined by the creation of grain micro- and submicrostructure. The increase in the width of the line from 11.6 to 31 × 10⁻³ rad is due to the appearance of second-type distortions and small regions of coherent scattering.

When the strain-hardened specimen was heated to room temperature there was a sharp reduction in hardness due to the change in crystal properties as a result of elevation of temperature. Here the difference in the hardness between the annealed and deformed specimens at - 180 and + 20°C remained the same, as the grain substructure created at -180° does not appear to undergo any change when heated to + 20°C. The reduction in the width of the interference lines when the specimens are heated from - 180 to + 20° is mainly due to reduction in secondtype distortions due to changes in crystal properties. In other words, second-type distortions which arise as a result of the strain-hardening of the specimen at - 180°C are partially relaxed when heated to room temperature and in this case there is a reduction in the elastic limit. A reduction in the width of the interference lines was observed in works [6, 7] when specimens of a number of different metals were heated to 20°C.

It is interesting to note that the second cooling of the deformed specimen to -180°C caused the hardness to increase once more from 98 to 220 HV despite the fact that part of the second-type distortions was relieved when the specimens were heated to 20°C .

The iron specimens also underwent deformation of 30% at $+20^{\circ}$ C. Hardness increased from 63 to 85 HV and the width of the interference lines from 11.0 to 19.8 × 10⁻³ rad (Fig. 3). When the deformed specimen was cooled to -180° C hardness increased to 200 HV. It is suggested that the strengthening of the iron at -180° C causes the creation of a more dispersed substructure in the metal than that

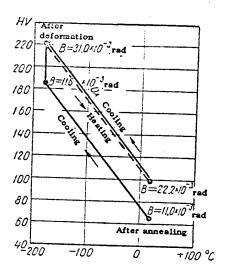


FIG. 1. Influence of deformation and temperature on hardness and the width of line (220) in iron. Deformation at low temperature.

which takes place after the same material has been strengthened at 20°C and this in its turn causes a greater strengthening effect.

A similar series of experiments was carried out on the nickel specimens. Unlike the iron, in annealed nickel hardness measured at -180° C was very little different from that measured at 20° C ($\Delta HV = 15$). When the nickel was deformed at -180° C there was a considerable strengthening effect. Hardness increased from 65 to 160 HV and the width of the lines from 11.4 to 23.9×10^{-3} . After heating to room temperature hardness decreased from 160 to 140 HV and the width of the line (222) remained practically unchanged. After a second cooling to -180° C hardness measured at -180° C was $160 \ HV$ (Fig. 4).

Thus, in the nickel specimen, in which the crystal properties change very little with reduction of temperature, second-type distortions arising as a result of strengthening at -180° C remain practically unchanged when heated to 20° C. As in the case of iron, when the nickel was deformed at 20° C the strengthening effect is less than with deformation at -180° C. The effect on fine structure and mechanical properties of cold plastic deformations at +20 and -180° C was also studied. Deformation at -180° C has a greater strengthening effect than at $+20^{\circ}$ C. The regions of coherent scattering are smaller with the low temperature deformation than after deformation at 20° C (Fig. 5).

Crystal properties and grain substructure: II

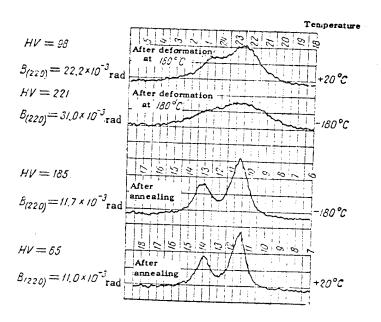


FIG. 2. Distribution curves for the intensity of line (220) in iron, obtained at two temperatures after annealing and after low-temperature deformation.

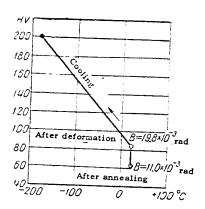


FIG. 3. Influence of deformation and temperature on hardness of iron. Deformation at room temperature.

From these experimental data it can be seen that the strain-hardened metals Fe and Ni behave differently with reduction of temperature. As was suggested, there is a sharp increase in the resistance of iron to plastic deformation at lower temperatures. Here it is the variation of crystal properties, in the case of strain-hardening at -180° C, which makes the greater contribution to the strength of the iron. Submicro-imperfections of the structure play a smaller part. Variations in the crystal properties of the iron cause an increase of 35 HV units in its

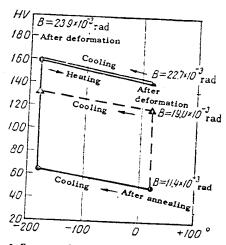


FIG. 4. Influence of deformation and temperature on hardness and width of line (222) in nickel.

hardness after deformation at -180°C. In nickel on the other hand, crystal properties play a much smaller part than that of the creation of submicrostructural imperfections.

The greater strain hardening effect observed after deformation at low temperatures appears to be due to the fact that at these temperatures conditions are favourable for the creation of a more dispersed grain submicrostructure. The fact that in iron, in which a sharp change of crystal properties is observed when the temperature is raised from

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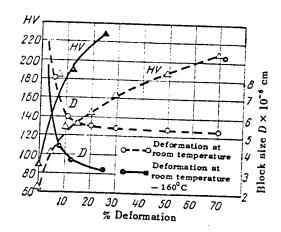


FIG. 5. Dependence of hardness and size of the regions of coherent scattering in nickel on the degree of deformation at various different temperatures.

-180 to + 20°C, there is also a reduction in second-type distortions (this is not found in nickel) is confirmed by the dependence of $\Delta a/a$ on crystal

properties.

Translated by V. Alford

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